PATENT SPECIFICATION

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(72) Inventors GERT HUMME, KARL DINGES, KARL-HEINZ OTT and HARRY ROHR



(54) HIGH-IMPACT NON-AGEING AES-POLYMERS

We, BAYER AKTIENGESELLSCHAFT, a body corporate organised under the laws of Germany, of 509 Leverkusen, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following 5 5 AES-Polymers are thermoplastic two-phase plastics materials in which acrylonitrile (A) and styrene (S) are graft polymerised onto an EPDM-rubber (E). EPDM-Rubbers are terpolymers of ethylene (E), propylene (P) and an unconjugated diene (D). AES-Polymers are basically known. They show improved stability to light and 10 10 weathering in comparison with corresponding ABS-graft polymers (styrene and acrylonitrile grafted onto butadiene polymers). Conventional processes for producing AES-polymers are technically involved and confined to special EP and EPDM-rubbers, in addition to which the resulting products are inferior to the corresponding ABS-polymers.

The processes normally used for producing AES-polymers are as follows: 15 15 Emulsion polymerisation. Styrene and acrylonitrile are polymerised in the presence of an EPDM-rubber emulsion or dispersion. However, as EPDM-rubbers are synthesised by solution polymerisation and not by emulsion polymerisation, the emulsions or dispersions have to be specially prepared. They show only limited stability in storage and the 20 20 grafting reaction is accompanied by heavy coagulation. Bulk or bulk-suspension polymerisation. EPDM-Rubbers are only soluble to a limited extent in mixtures of styrene and 25 acrylonitrile. Accordingly, auxiliary solvents have to be added, separately pre-25 pared graft polymers have to be introduced and/or special programmes have to be maintained in regard to temperature control and addition of the monomer mixture. Solution or solution-precipitation polymerisation. Although the processes which have hitherto been proposed in this respect are technically feasable, they either involve considerable technical outlay, such as 30 30 those described in DT—OS 1,745,945 (multistage process, peroxide)
DT—OS 1,769,052 (solvent separation by distillation, temperature and metering programmes)
DT—OS 2,262,239 (pressure apparatus) 35 DT—OS 2,302,014} (solvent combinations) 35 DT-OS 2,304,377 involve a high safety risk, such as those described in DT-OS 2,216,845 (decomposition temperature of the activator) are confined to special EPDM-rubbers, such as those described in DT—OS 1,769,052 or cannot be adequately reproduced, such as those described in DT—OS 1,745,945 (peroxide formation).

A solution polymerisation process for the production of AES-polymers has 40 40 now been found which does not have any of the disadvantages referred to above and which, at the same time, yields products which are superior in their combinations of properties to standard commercial-grade ABS-polymers. 45 45

	The present invention provides a non-ageing AES polymer moulding composition comprising:	
	A) 10 to 90 parts by weight of a graft copolymer produced by the graft polymerisation of styrene or of styrene and acrylonitrile (the styrene	
5	in each case being completely or partly replac able by α -methyl styrene) in a ratio by weight of from 4:1 to 3:2, as graft monomer, in the presence of an EPDM rubber or of a mixture of an EPDM	5
	rubber with a saturated rubber, as graft base, the polymerisation being carried out in an aromatic optionally halogen-substituted	
10	hydrocarbon or in a mixture of such aromatic hydrocarbons as solvent at a temperature of above 80°C, and the following ratios by weight being maintained:	10
•	solvent 2.5.1 to 10.1	•
	graft monomer + graft base	• ÷
	graft base	
	graft monomer = 1:3 to 4:1, and	
15	B) 90 to 10 parts by weight of a styrene homopolymer or of a copolymer of a styrene with acrylonitrile or methacrylonitrile (the styrene in each case optionally being side-chain-substituted or nuclear-substituted) in a ratio by weight of from 80:20 to 20:50	15
20	The invention also provides a process for the production of a moulding composition as defined above, which comprises mixing the polymers A) and B) as solids or in solution.	20
	A. Graft polymers.	•
	A.1 EPDM-Rubber. "EPDM-Rubbers" in the context of the invention are copolymers of ethylene,	•
25	propylene and an unconjugated diene. The ratio by weight of ethylene to propylene is preferably from 75:25 to 40:60. The diene is incorporated in the copolymer in such quantities and in such form that iodine numbers of from 2 to 30 are obtained, corresponding to approximately 1—15 C=C bonds per 1000 carbon	25
30	atoms. The monomer units may be arranged either statistically or in blocks. Instead of one diene, mixtures of several dienes may also be incorporated in	30
	the copolymer. Mixtures of an EPDM-rubber with other rubbers containing less than 50 C=C bonds per 1000 carbon atoms may also be used in accordance with the invention.	
35	Basically any unconjugated dienes may be used as comonomer. Preferred dienes are dicyclopentadiene, 1,4-hexadiene and 5-ethylidene-2-norbornene. It is also	35
	possible to use their homologues such as other alkylidene norbornenes, tricyclopentadiene, heptadiene or octadiene.	
	EPDM-Rubbers with a Mooney plasticity (ML 1 + 4', 100°C) of from 20 to 150 are particularly preferred.	
40	A.2 Graft monomers. Graft monomers in the context of the invention are styrene and mixtures of	40
	styrene and acrylonitrile in a ratio by weight of from 4:1 to 3:2. The styrene may be completely or partly replaced by α -methyl styrene.	7
45	The ratio by weight of graft base to graft monomer is from 1:3 to 4:1, preferably from 1:2 to 2.4:1.	45
	A.3 Graft polymerisation. Graft polymerisation means polymerisation of the graft monomers in the	
50	presence of the graft base. Part of the monomers is polymerised onto the graft base in the form of side chains. This part of the polymer formed is chemically attached	50
30	to the graft base. In addition, free polymer of the graft monomers is formed. The ratio by weight of the polymer chemically attached (grafted) to the graft base to the free polymer is known as the degree of grafting. This degree of grafting and the molecular weight both of the grafted polymer and of the free polymer can be	

	1,307,030	3
5	influenced to a considerable extent by the polymerisation conditions (temperature, activation, molecular weight r gulation, solvent, stirring conditions, quantities of monomer). Accordingly, the polymerisation conditions have to be carefully selected to obtain products with optimum properties. The polymerisation reaction is initiated by radical activators. The polymerisation activator, polymerisation velocity and polymerisation temperature are interrelated. For a given polymerisation temperature, it is possible to select suitable activators on the basis of the half lives tabulated in the	5
10	Temperatures of above 80°C, preferably in the range of from 100 to 140°C and, with particular preference, in the range of from 110 to 130°C are required for the graft polymerisation reaction	10
15	Peroxide activators, such as di-tertbutyl peroxide, cumene hydroperoxide, tertbutyl hydroperoxide and dicumyl peroxide are particularly suitable. In cases where reducing agents are added, it is also possible to use activators of which the rate of decomposition at the temperatures indicated is basically too low. Suitable molecular weight regulators are relatively long-chain mercaptans, terpinolenes and α -olefins.	15
20	A.4 Solvents.	
25	In principle, it is possible to use any of the usual aromatic hydrocarbons and their halogen derivatives as solvents. Examples of suitable solvents are benzene, toluene, ethyl benzene, xylene, trimethyl benzene, cumene and chlorobenzene. Benzene and toluene are preferably used as the solvents by virtue of their minimal regulator effect (transfer reactions). In cases where these solvents	20
	used, the products according to the invention are obtained when the ratio by weight of solvent to graft monomer + graft base is from 2.5:1 to 10:1. This ratio is preferably <10 for benzene and preferably <5 for toluene.	25
30	B. Styrene, styrene-acrylonitrile copolymers ("SAN-copolymer"). The styrene polymers and styrene-acrylonitrile copolymers (providing they are not formed during the graft polymerisation reaction) may be produced by any of the polymerisation processes normally used for this purpose. The styrene may be completely or partly replaced by a side-chain-substituted or nuclear-substituted styrene.	30
35	The SAN-copolymer preferably contains styrene and acrylonitrile units in a ratio by weight of from 80:20 to 60:40. Where the styrene is replaced by α -methyl styrene, copolymers with a ratio by weight of α -methyl styrene to acrylonitrile of 69:31 have proved to be particularly favourable.	35
40	In the monomer combination of styrene and acrylonitrile, the styrene may also be partly replaced by other monomers, or in conjunction with other monomers the acrylonitrile content may be increased to beyond 60% by weight. Examples of SAN-copolymers modified in this way are terpolymers of styrene and acrylonitrile with methyl methacrylate, and terpolymers of styrene and acrylonitrile with α -olefins.	40
45	The styrene may also be replaced by α -methyl styrene or by other nucleus- or side-chain-substituted styrenes, whilst the acrylonitrile may also be replaced by methacrylonitrile. The SAN-copolymer may be produced by different mathyl.	45
50	active substances such as, for example, alkyl sulphates, alkyl sulphonates, aryl alkyl sulphonates, the alkali metal salts of saturated or unsaturated fatty acids and the alkali metal salts of disproportionated or hydrogenated abietic or tall oil acids. Suitable activators are standard organic and increasing	50
55	agent and a reducing agent, heavy metal ions additionally being present in the reaction medium. The molecular weights may be adjusted with so called a selection in the selection.	55
60	regulators, primarily relatively long-chain mercaptans or terpinolene. Regulation is also possible by adding α-olefins. In cases where polymerisation is carried out in solution or in bulk, it is possible to use aromatic hydrocarbons or the monomers themselves as solvents; suitable activators are organic peroxides or azo compounds. In cases where in this process polymerisation is only continued up to a certain	60

5	solid polymer, fo the case of emul In cases w suspension poly	unreacted monomers and the solvent may be removed from the r example by concentration through evaporation in a screw or, in Ision polymerisation, even through a thin layer evaporator. There the styrene-acrylonitrile copolymers are produced by merisation, the usual dispersants, such as polyvinyl alcohol or sed polyvinyl acetate, may be used as suspension stabilisers.	5
10	Graft copoly (precipitation, st	graft copolymer A. ymer (A) may be isolated from its solutions by the usual methods ripping or concentration through evaporation in the screw). It is erred to use stripping and to remove the solvent by evaporation	10
15	The solid portion for example musclutions of the contain from 10 (B), preferably freight of (B).	graft polymer A and the styrene polymer B. blymers may be mixed by means of standard compounding units, ltiroll stands, mixing extruders or internal kneaders. However, polymers may also be mixed and worked up. The mixture should to 90 parts by weight of (A) and from 90 to 10 parts by weight of from 10 to 60 parts by weight of (A) and from 90 to 40 parts by the mixture preferably has an EPDM-rubber content of ≤30% by	15
20	several such graf the limits specific base used may b		20
25	mixtures during of this kind inclu static agents, lub Stabiliser co	es normally used for moulding compositions may be added to the their working up, further processing and final shaping. Additives ude antioxidants, UV-stabilisers, hydroperoxide neutralisers, antioricants, flameproofing agents, fillers and pigments. Demonstrations of light stabilisers of the methyl piperidine type and	25
30	be carried out in moulding, sheet the extrusion of	the benztriazole type are particularly suitable. Final shaping may the usual processing units and includes, for example, injection extrusion with subsequent vacuum forming, cold forming of films, tubes and profiles, and calender processing. rwise stated, the parts quoted in the Examples are parts by weight.	30
35	General recipe: E parts by weight L parts by weight	E. EXAMPLES. of the EPDM graft polymers. ht of EPDM rubber are dissolved in ht of solvent.	35
40	A parts by weig T°C (= polymeri initiator, polyme T hours at the a	nt of styrene and ht of acrylonitrile are added and the solution is heated to sation temperature). Following the addition of J parts by weight of crisation is carried out for bove-mentioned polymerisation temperature.	40
45	added to it, bas antioxidant (2,6- (dilauryl thiodip: crumbs obtained	onversions obtained amount to >98%. The polymer solution has sed on the sum: $E + S + A$, 0.5 part by weight of a phenolic di-tert-butyl-p-cresol) and 0.5 part by weight of a co-stabiliser ropionate), and the polymer product is isolated by stripping. The diare dried at 70°C in a vacuum drying cabinet.	45
50	material:	V2A steel	50
	pressure load:	up to 6 bars	
	stirring:	wall-sweeping stirrer designed for viscosities of ≥1000 poises	:
	stripper:	standard stripper of the kind used in rubber technology	
55	The polym	rylonitrile copolymers. erisation process used to produce the styrene-acrylonitrile	55

	products and also specially polymerised products are used for producing the mixtures in the following Examples.	
5	 The following measurable values are used for characterising the products: acrylonitrile content; calculated by N₂-elemental analysis styrene or α-methyl styrene content; calculated as 100%-% acrylonitrile, Mn = number average molecular weight determined by osmotic measurements, 	5
	 4. Mw = weight average molecular weight determined by light scattering, Mw 5. U = 1 (molecular inconsistency) 	
10	 6. Mη = molecular weight from viscosity measurements (dimethyl formamide, 20°C) 	10
15	E.3 Production of the polymer mixture. X parts by weight of the EPDM graft polymer according to specification (E.1) and Y parts by weight of the copolymer according to specification (E.2) are compacted into a rough sheet on a two-roll stand in the presence of 0.5 part by weight of the bis-stearylamide of ethylene diamine. The rough sheet is granulated and standard small test bars are produced from the granulate by injection moulding at a melt temperature of 220°C.	15
20	E.4 Test methods. Impact strength (a _k) and notched impact strength (a _k) at room temperature and at -40°C (DIN 53 453, unit: kJ/m²). Vicat softening temperature (53 460), (°)	20
25	Ball indentation hardness H _c , 30 secs. (DIN 53 452), (MPa) Ageing test: Weather-O-meter of Messrs. Atlas Electro, Chicago. Impact strength is measured in dependence upon the ageing time. Thermoplastic processability: H. Ebneth, K. Bohm, "Fliessfahigkeit von ABS-Polymerisation mit hilfe einer neuartigen Flachspirale", (Fluidity of ABS Polymers by means of a Novel Flat Spiral), Plastverabeiter 19 (1968), 4, pages 261—269; test temperature 220°C (cm)	25
30	EXAMPLES 1—5. (Variation of the EPDM Rubbers as Graft Base). The EPDM graft polymers are produced in accordance with general	30
35	specification E.1. L = 500 parts by weight of benzene E = 43 parts by weight of EPDM rubber, a more detailed characterisation of the rubber being given in Table 1 S = 43 parts by weight of styrene A = 14 parts by weight of acrylonitrile	35
40	T = 120°C J = 0.9 part by weight of di-tert-butyl peroxide t = 14 hours	40
45	The polymer blends are produced in accordance with general specification E.3 with X = 40 parts by weight of EPDM graft polymer Y = 60 parts by weight of styrene-acrylonitrile copolymer The styrene-acrylonitrile copolymer is characterised by the following data in accordance with specification E.2:	45
	acrylonitrile content: 26.2% by weight styrene content: 73.8% by weight	
50	Mη: 120,000 U: 2.1	50

	5	EN,S	20	37	23.3	77.1	92
	4	ХН	13	144	18.9	76.7	94
	3	DCPD	11	70	25.9	72.2	95
1 H	2	EN	27	24	25.4	72.5	91
TABLE 1	1	EN	23	99	24.6	72.5	93
	Example	Diene component*) of the EPDM rubber	lodine number	Mooney-value (ML 1 + 4', 100°C)	$a_{\rm k}$, 20° C (kJ/m ³)	H _c , 30 sec. (M Pa)	Vicat temperature (°C)

*) EN = 5-ethylidene-2-norbornene

DCPD = dicyclopentadiene

HX = 1,4-hexadiene

sequence polymer

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Further properties of the product produced in accordance with Example 3 and of a standard commercial-grade ABS polymer are compared in Table 2.

TABLE 2

	· · · · · · · · · · · · · · · · · · ·		Example 3	ABS
a _n , 20°C (k	cJ/m²)	٠	ub *)	ub
a _n , 0°C (,,)		ub	ub
a _n , -20°C(,,)		ub	ub -
a _n , -40°C(,,)		ub	73
a _k , 20°C (,,)		24.6	12.1
H _c , 30 sec.	(M Pa)		72.5	. 85
Vicat (°C)			93	95
Processibili	ity (cm)		50	44
Ageing test				
$a_n (kJ/m^2)$	after 0	(h)	ub	ub
. 91	after 100	(h)	ub	ub
***	after 300	(h)	ub	54
,,	after 500	(h)	ub ·	34
•	after 800	(h)	ub	10
,,	after 1200	(h)	ub	

^{*)} ub = of 10 test specimens, more than 50% remained unbroken during impact strength measurement.

Table 1 shows that products of comparable mechanical properties are obtained irrespective of the type of EPDM rubber used as graft base. In addition, Table 2 shows that these products are distinctly superior to the standard commercial grade ABS products in regard to toughness, processibility and, above all, weathering resistance.

EXAMPLES 6 and 7.

The basic recipe of Examples 1 to 5 is used. It is modified in regard to the type of rubber used (EPDM rubber based on dicyclopentadiene; iodine number: 20; Mooney viscosity ML 1 + 4', 100°C: 28) and the solvent L. Table 3 shows that products with comparable properties are obtained both in benzene and in toluene. 10

10

5

TABLE 3

Examples	6	7
Solvent	benzene	toluene
quantity of solvent (L) (parts by weight)	500	250
a _k , 20°C (kJ/m ²)	25.3	27.6
H _c , 30 seconds (M Pa)	77.7	73.6
Vicat temperature (°C)	93	94

EXAMPLES 8 and 9.

Examples 6 and 7 are repeated with the sole difference that the quantity of solvent is doubled.

TABLE 4

Examples	8	9.
solvent	benzene	toluene
quantity of solvent (L) (parts by weight)	1000	500
a _k , 20°C (kJ/m²)	8.2	1.9
H _c , 30 seconds (M Pa)	64.0	63.2
Vicat temperatures (°C)	91	89

EXAMPLES 10 to 15.

The graft polymer of styrene-acrylonitrile on an EPDM rubber with dicyclopentadiene as graft base, described in Example 3, was used for the tests summarised in Table 5. The quantitative ratio of graft polymer (X) and resin component (Y) and also the chemical structure of the resin component were varied. The polymer blend was produced under the conditions specified in E.3.

10

		TABLE 5	-			
Examples	10	11	12	13	14	15
Characterisation of the resin component						
% acrylonitrile	26.2	26.2	32.2	27.1	29.1	28.0
% styrene	73.8	73.8	67.7	72.9	į	ſ
% $lpha$ -methyl styrene	ſ	. 1	t		70.9	72.0
Mη	120,000	120,000	000'06	125,000	000'09	160,000
Ω	2.1	2.1	1.9	3.8	2.0	1.8
X (% by weight)	. 30	20	40	35	40	40
Y (% by weight)	70	80	09	65	09	09
ak, 20°C (kJ/m²)	19.5	11.1	20.0	18,1	15.5	24.0
H _c , 30 seconds (M Pa)	99.5	110.4	65.3	83.9	63.9	62.7
Vicat temperature (°C)	96	76	66	96	104	105

EXAMPLES 16, 17 and Comparative Example 1. The procedure of Example 1 is repeated with the changes indicated in Table 6 regarding temperature (T), activator (J) and polymerisation time (t).

	TABLE 6		
Examples	16	17	Comparative Example 1
polymerisation temperature (T) (°C)	130	110	80
activator (J)	di-tert butyl peroxide	di-tert butyl peroxide	benzoyl peroxide
parts by weight of activator (J)	6.0	6.0	1.5
polymerisation time (t)	10	20	18
a_k , $20^{\circ}C$ (kJ/m²)	13.1	26.2	2.6
H _c , 30 seconds (M Pa)	75.9	73.9	71.0
Vicat temperature (°C)	92	92	91

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EXAMPLES 18 to 20.

The test specimens are produced in accordance with Example 3, the only difference being that the ratio of graft base to graft monomers and the mixing ratio of graft polymer to resin component (X:Y) are varied. The mixing ratio of graft polymer to resin component is selected in depend nce upon the graft polymer in such a way that the polymer blend has a constant EPDM rubber content of 17.2%.

5

·	TABLE 7		•
Example	18	19	20
E (parts by weight)	50	60	70
A (parts by weight)	12.5	10.0	7.5
S (parts by weight)	37.5	30	22.5
X (parts by weight)	34.4	28.6	24.6
Y (parts by weight)	65.6	71.3	75.4
a _k , 20°C (kJ/m²)	30.7	11.0	3.8
H _c , 30 seconds (M Pa)	75.9	76.2	78.0
Vicat temperature (°C)	94	96	93

EXAMPLES 21—23 and Comparative Example 2.

Grafting onto rubber mixtures of EPDM rubber and ethylene-vinyl acetate

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The tests summarised in Table 8 are based on the same specification as Example 19 with changes to a) the type of rubber used as graft base, but not the total quantity of rubber used, EPDM rubber: diene component: 5-ethylidene-2-norbornene

iodine number: 24 Mooney viscosity ML 1 + 4', 100°C:95

15

ethylene-vinyl acetate number:

vinyl acetate names:

vinyl acetate content: 45%

Mooney viscosity ML 1 + 4', 100°C:20

and b) the mixing ratio of EPDM graft polymer (X) to styrene-acrylonitrile copolymer

X = 40 parts by weight

Y = 60 parts by weight

20

TABLE 8

Examples	21	22	23	Comparative Example 2
parts by weight of EPDM rubber	50	45	25	- .
parts by weight of EVA rubber	_	5	25	50
a_k , 20°C (kJ/m²)	37.0	37.0	29.1	5.5
H _c , 30 seconds (M Pa)	64.9	62.9	62.8	64
Vicat temperature (°C)	93	93	92	90

The tests show that ≥50% of other saturat d rubbers may be added to the EPDM rubbers without producing any significant change in the level of properties. **EXAMPLE 24.** The EPDM graft polymers of Example 21 and Example 20 are mixed in the following quantitative ratio with the styrene-acrylonitrile copolymer of Examples 1 5 to 5 in accordance with specification E.3 for the polymer blend:
4.9 parts by weight of EPDM graft polymer of Example 20
27.5 parts by weight of EPDM graft polymer of Example 21
67.6 parts by weight of styrene-acylonitrile copolymer of Examples 1 to 5 The product has the following mechanical characteristics: 10 a_k, 20°C (kJ/m²): 30.8 H_e, 30 seconds (M Pa): 72.8 Vicat temperature (°C): 93 EXAMPLE 25. Example 1 was repeated with the difference that, instead of being isolated by 15 15 stripping, the graft polymer was isolated in an evaporation screw. a_k, 20°C (kJ/m²): 23.6 H_p, 30 seconds (M Pa): 74.5 Vicat temperature (°C): 91 20 WHAT WE CLAIM IS:-20 1. A non-ageing AES polymer moulding composition comprising: A) 10 to 90 parts by weight of a graft copolymer produced by the graft polymerisation of styrene or of styrene and acrylonitrile (the styrene in each case being completely or partly replaceable by α -methyl styrene) in a ratio by weight of from 4:1 to 3:2, as graft monomer, in the presence of an EPDM rubber or of a mixture of an EPDM 25 25 rubber with a saturated rubber, as graft base, the polymerisation being carried out in an aromatic optionally halogen-substituted hydrocarbon or in a mixture of such aromatic hydrocarbons as solvent at a temperature of above 80°C, and the following ratios by 30 30 weight being maintained: solvent = 2.5:1 to 10:1graft monomer + graft base graft base = 1:3 to 4:1, and graft monomer B) 90 to 10 parts by weight of a styrene homopolymer or of a copolymer of a 35 styrene with acrylonitrile or methacrylonitrile (the styrene in each 35 case optionally being side-chain-substituted or nuclear-substituted) in a ratio by weight of from 80:20 to 50:50. 2. A moulding composition as claimed in claim 1, wherein the graft polymerisation reaction is carried out in benzene or toluene as the aromatic 40 hydrocarbon. 3. A moulding composition as claimed in claim 2, wherein the quotient solvent graft monomer + graft base is <10 for benzene and <5 for toluene. 4. A moulding composition as claimed in any of claims 1 to 3, wherein the diene component of the EPDM rubber is 5-ethylidene-2-norbornene, dicyclopentadiene or 1,4-hexadiene. 45 45 5. A moulding composition as claimed in any of claims 1 to 4, wherein the styrene of the homopolymer or copolymer of component B) is styrene or α -methyl styrene.

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6. A moulding composition as claimed in claim 1, substantially as herein described with reference to any of the specific Examples.

7. A process for the production of a moulding composition as defined in claim 1, which comprises mixing the polymers A) and B) as solids or in solution.

8. A process as claimed in claim 7, substantially as herein described with ref rence to any of the specific Examples.

9. A moulding composition when produced by a process as claimed in claim 7 or 8

ELKINGTON & FIFE, Chartered Patent Agents, High Holborn House, 52/54 High Holborn, London WC1V 6SH. Agents for the Applicants.

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